# U.S. Air Force Update on Replacement of Hexavalent Chrome Coatings

2008 International Workshop on Pollution Prevention and Sustainable Development 18-20 November 2008



**Bill Hoogsteden** 

AFRL/RXSSO

937-656-4223



william.hoogsteden@wpaf.af.mil



## **Presentation Outline**



- Background
  - Integration plan

- Mg Rich primer work
- Sicopoxy primer work

- Electroactive polymer work
  - BAM-PPV



# AFRL Chromium-free Coating Systems: Integration Plan



#### Scope, Definitions, and Goal ......

Chromium is a HazMat and must be eliminated

System Schematic - Not to Scale



Specialty coatings not considered in this plan at this time

# AFRL Coatings System Performance Approach

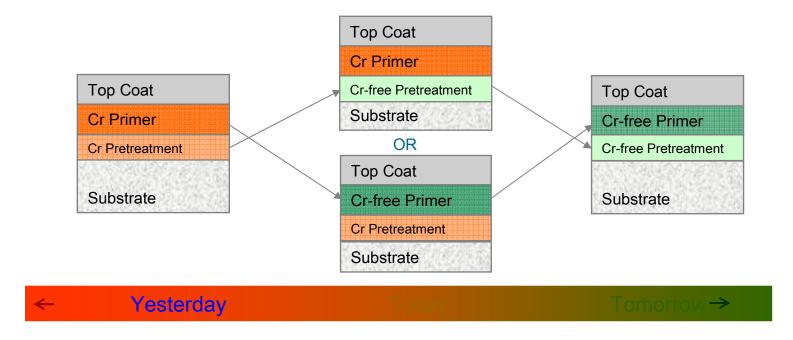
- Stack up meets all the performance standards for current aircraft exterior (OML) coating systems
- Performance of entire system evaluated, not individual components
- Not controlled by individual component specifications
- Focus on a *totally* chromium-free system
- Application methods are user-friendly, do not affect productivity/scheduling, and are able to be applied to any size aircraft



# AFRL Chromium-free Coating Systems Integration Plan



No qualified, completely chromium-free system yet ...



- ➤ Some individual chromium-free components cannot be combined with some MIL SPEC or other components; e.g., PreKote with Deft 02-GN-084
- ➤ AFRL currently testing two chromium-free coating systems that show most promise based on screening/performance testing



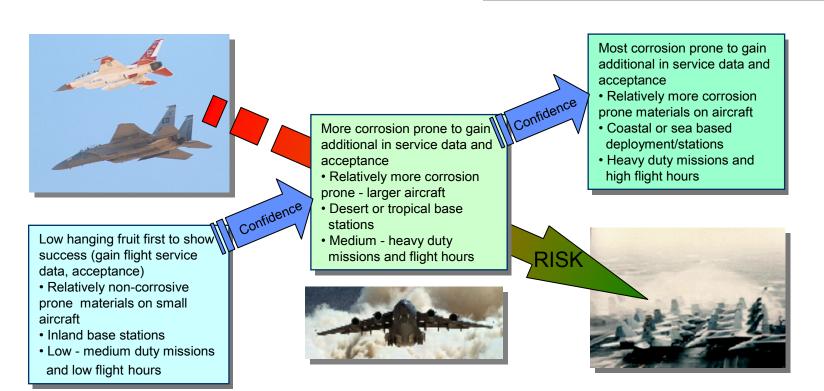
# AFRL/RX Approach to Corrosion Control and Prevention



- **Step 1:** Find a solution that meets or exceeds performance requirements, including materials compatibility, corrosion resistance
- Step 2: Minimize risk of implementation by gradually expanding laboratory and field tests by size of aircraft, corrosiveness of materials on aircraft, and aggressiveness of service environment

#### Materials Compatibility Testing

- Immersion in working, maintenance, repair fluids Corrosion Testing
  - · Electrochemical open circuit, polarization, impedance
  - Salt Fog Exposure (ASTM B 117)
  - Alternating Immersion (ASTM G 85)
  - Salt Fog + SO<sub>2</sub> (ASTM G 87; Navy requirement)
  - Cyclic Exposure (GM 9540P)
  - Hydrogen Embrittlement (ASTM F 519, F 1624)
  - Hydrogen Re-embrittlement (ASTM F 519, F 1624)
  - Stress Corrosion Cracking (ASTM F 1624)





# AFRL Chromium-free Coating Systems Integration Plan



#### Current AFRL approach .....

- □ Obtain buy in by all stakeholders before spending funds
- □ Perform additional performance testing with first system identified
  - ✓ Alodine 5200/5700 Sicopoxy 577-630 MIL-PRF-85285 (Deft 03-GY-001)
  - ✓ Test other Cr-free pretreatments for compatibility with primer
  - ✓ Investigate further effect of topcoat on corrosion resistance
  - ✓ Perform field (outdoor) exposure tests at a coastal site
  - ✓ Coordinate a limited flight test with a T-38 at Randolph AFB
  - ✓ Based on test results, decide on next step
- Perform additional testing with second system identified
  - **✓ PreKote** Mg-rich Primer MIL-PRF-85285 (Aerodur 5000)
  - ✓ Investigate further effect of system components on corrosion resistance
  - ✓ Perform field (outdoor) exposure tests at a coastal site
  - ✓ Coordinate limited flight tests with a F-16 at Hill AFB and other aircraft
  - ✓ Based on test results, decide on next step
- □ Coordinate testing where possible with other projects/programs
- Use risk reduction approach
- □ Communicate results to all interested parties: solicit feedback



# AFRL Chromium-free Coating Systems Integration Plan



#### Benefits/Impacts

- □ Elimination of HazMats in paint coatings
  - ➤ Meets requirements of Executive Orders 13148/13423
  - ➤ Helps compliance with new OSHA Cr PEL
  - Reduces worker exposure to known carcinogen
- □ Materials substitution only no capital funds required; no new equipment needed; no special worker training
- □ Aircraft performance in service not affected
  - Flight performance not compromised
  - Same maintenance, de-icing, etc. chemicals can be used
- □ Fieldability world wide (compliance with foreign environmental regulations)
  - Supports mission sustainability and readiness





# Mg Rich Primer



## Mg-rich Primer – What is it?



# APC Grade Topcoat Mg-rich Primer (~1 mil thick) Alguage (~2 kg)

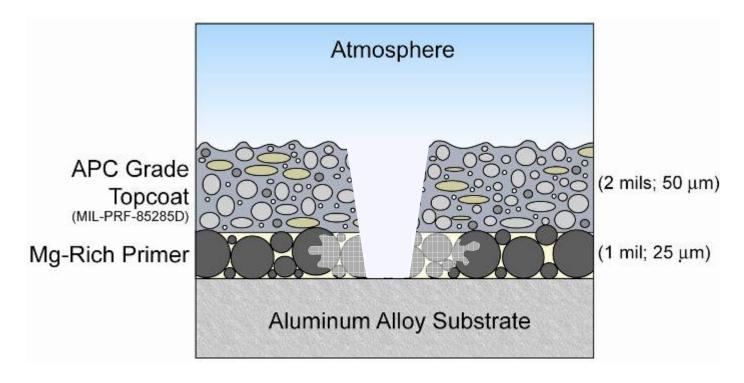
- Developmental non-Cr primer as part of a fully non-Cr coating system
- Concept developed by NDSU
- Licensed & product development by a major aerospace coating company
- Provides "cathodic" corrosion protection of the substrate
  - analogous to a Zn-rich primer for steel
  - does not use inhibitors
- NEW TECHNOLOGY = NEW RULES; still some unknowns, may require special considerations
- Designed as "Drop In" for MIL-PRF-23377
- Currently used with PreKote.



## How does it work? - Mg-Rich Primer



 <u>Prevention</u>: provided by barrier properties of the primer and topcoat (conventional); both Mg pigment and substrate protected



 Control: Mg particles are more active than the aluminum; the pigment corrodes and the substrate doesn't



# **TECHNICAL APROACH (cont.)**



- Leverage previous OSD sponsored material development project
  - Resulted in commercially viable primer formulation from Akzo-Nobel Aerospace Coatings;
     Aerodur 2100 product
- Project coordination with target aircraft corrosion managers to define operational requirements and "go / no-go" decision points
  - F-16; Shane Kirby & Glen Baker (Hill AFB)
  - C-130; Molly Statham (Robins AFB)
  - F/A-18; Craig Matzdorf (Pax River NAVAIR)
  - HH-60; LCDR Mark Ward (Elizabeth City Coast Guard)
- Develop comprehensive test plan for laboratory qualification of Mg-rich primer technology
  - MIL-PRF-23377J (primer spec for general T.O. 1-1-8 applications & NAVAIR use)
    - + SO2 Salt Fog for NAVAIR
  - MIL-PRF-32239 (new coating system specification for Air Force use)
- Develop additional "beyond/outside spec" evaluations vetted through corrosion managers
  - Emphasis on data relevant to weapons system managers (e.g., environmental conditions, pot life windows, cure time windows, repairability, etc.)



# **TECHNICAL APROACH (cont.)**



- Generate samples and perform qualification and "beyond spec" experiments utilizing permutations of Mg-rich and Cr coating systems
  - Only one Mg-rich primer evaluated:
    - Aerodur 2100
  - Three "APC" grade topcoats in Fed. Std. 36173 color evaluated:
    - Defthane ELT (Deft)
    - Aerodur 5000 (Akzo-Nobel)
    - PRC 9311 (PRC-DeSoto)
  - Two "specialty" coatings evaluated:
    - F-16 topcoat
    - F-16 mid-coat
  - Two chromate control primers evaluated:
    - 02-GY-40 (Deft)
    - PRC CA7233 (PRC-DeSoto)
- Outdoor Performance Evaluation



**Daytona Beach outdoor exposure evaluation** 

Early flight demonstration on Coast Guard HH-60 panels



# **TECHNICAL APROACH (cont.)**



- Submit samples and obtain certification by Air Force fire safety office regarding flammability and approved fire control methods
- Presentation of technical data package to weapon system corrosion managers for approval of flight demonstration
- Coordination and application of Mg-rich coating system onto selected aircraft (½ Mg-rich + ½ Cr control)
  - Performed at depot location; oversight by CTIO
- Periodic performance evaluation of selected aircraft
  - Minimum of every 6 months
  - Out-years responsibility of corrosion managers
- Modification of AF T.O.'s by AF/CPCO
  - Results in approved use for specific aircraft
- NSN's assigned for Aerodur 2100 product
- Final Report







- Completed qualification and integration test plan with coordination of corrosion managers
  - Qualification to "system level" spec MIL-PRF-32239
  - Qualification to MIL-PRF-23377J + SO<sub>2</sub> for NAVAIR
  - Performance evaluation with various fasteners
  - Reparability of Mg-rich coatings
  - Performance over alternative substrates not in MIL-SPEC
  - Flight Demonstration target aircraft
    - F-16 (Air Force)
    - C-130 (Air Force)
    - P-3 (Navy)
    - HH-60 (Coast Guard)





# Mg Rich Primer Status

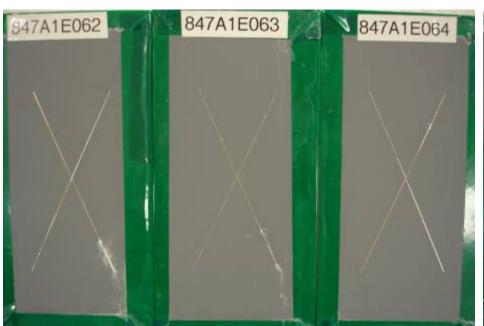


- Round Robin Mg Rich Primer B-117 Test
  - 5 Organizations
  - Statistical Performance B-117 Data
- Outdoor Exposure
  - Daytona Beach
  - KSC
- MIL-PRF-32239 Qualification if testing is successful





#### Mg-Rich + Aerodur 5000 650 hrs ASTM B 117 Salt Fog





2024-T3 7075-T6

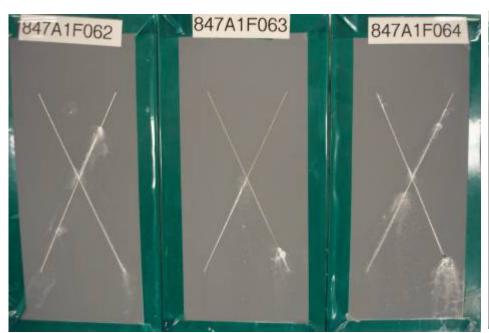


Results not consistent with prior testing!





# Mg-Rich + Defthane ELT 650 hrs ASTM B 117 Salt Fog





2024-T3 7075-T6

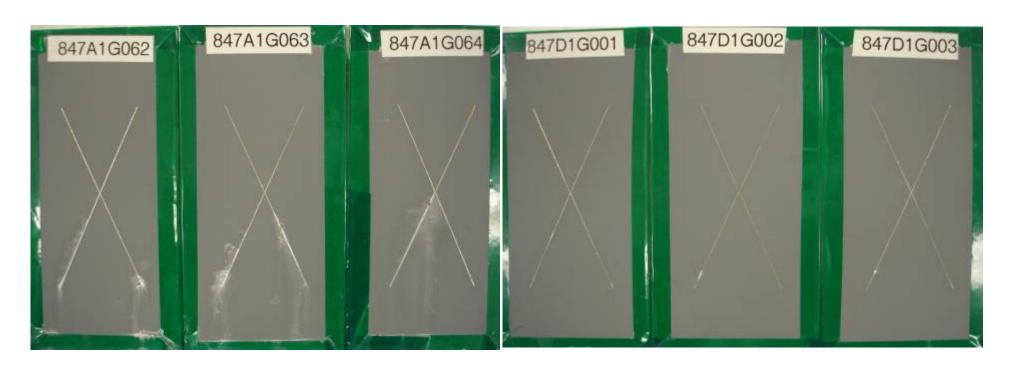
Results not consistent with prior testing!







Mg-Rich + PRC 9311 650 hrs ASTM B 117 Salt Fog



2024-T3 7075-T6









- Panels pulled at 650 hrs. due to testing anomaly
  - Except those with F-16 system
  - Not a chamber issue because Cr controls look good
- Most probable reason is the back sides were inadvertently left "bare"
  - Previous test always sealed edges and backs
  - Mg-rich primer works via cathodic protection, if an electrolyte bridge between the back side and the front is formed (i.e., the panel is completely "wet") the exposed area now includes the open back side
- Round Robin test plan developed and currently being worked to resolve the issue
  - Same lot of primer testing well at Akzo-Nobel with edges and back sides sealed







- Round Robin Testing
- Variables
  - Source of Aluminum alloy (AFRL, ANAC)
  - Panel preparation (AFRL, ANAC)
  - ASTM B 117 chamber (AFRL, ANAC, NAVAIR)
  - Sealed versus unsealed
- Constants
  - Formulation and lot number of Mg-rich primer
  - Formulation and lot number of PreKote
  - Formulation and lot number of topcoat
  - Scribe cutting (AFRL)
  - Sealing of panels







F-16 Coating System on 2024-T3 1,835 hrs ASTM B 117 Salt Fog



Chromate Control (severe blistering; poor scribe)

Mg-Rich System (no blistering; much better than control)





F-16 Coating System on 7075-T6 1,835 hrs ASTM B 117 Salt Fog





Chromate Control (severe blistering; poor scribe)

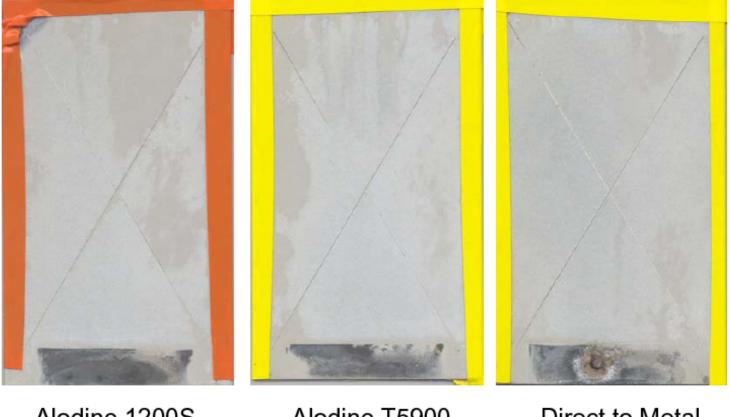
Mg-Rich System (no blistering; much better than control)





#### NAVAIR Testing in ASTM B 117 (sealed)

Figure 8: XP406 on 2024-T3 Aluminum after 2160 hrs (90 days) of ASTM B 117 Neutral Salt Fog





Alodine 1200S

Alodine T5900

Direct to Metal





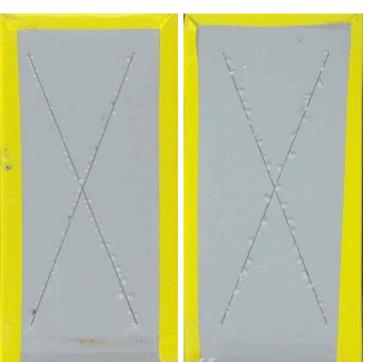
#### NAVAIR Testing in SO<sub>2</sub> Salt Fog and Compatibility with TCP

XP417 Mg-Rich System MIL-PRF-23377 Chromated Control

AA 2024-T3 AA 7075-T6

AA 2024-T3

AA 7075-T6









1,050 hrs. of SO<sub>2</sub> Salt Fog







# 6 Months Exposure at Daytona Beach

Excellent corrosion protection with bright shiny scribes on both Cr control and Mg-rich samples

Corrosion starting to show evidence on "blank" negative controls



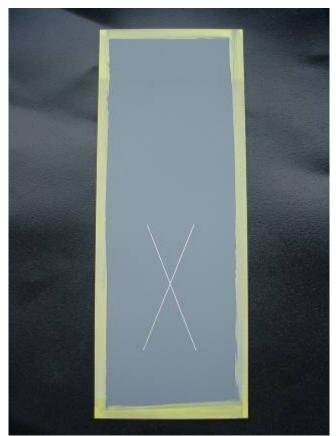




## 6 Months Daytona Beach Exposure



- PreKote™
- Mg-rich primer
- APC grade MIL-PRF-85285D topcoat



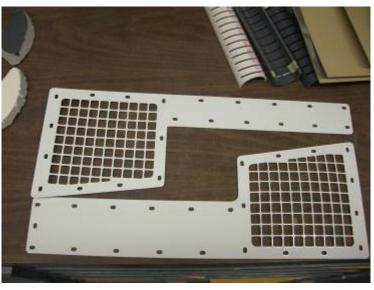
- CCC
- MIL-PRF-23377J Primer
- APC grade MIL-PRF-85285D topcoat

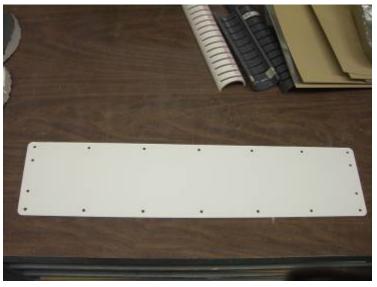












- Coast Guard HH-60 panels painted with Mg-rich primer and gloss white topcoat
- Samples to be monitored for corrosion and gloss retention
- Sent to Coast Guard and put in service in December 2007







# **SICOPOXY WORK**



# AFRL Non-Chrome Coatings Evaluation: Brief History/Overview



#### Phase IV Test Results - Neutral Salt Fog Exposure

System T (Alodine 5200/Sicopoxy 577-639/Deft 03-GY-321)
 performed best compared to Cr-containing controls

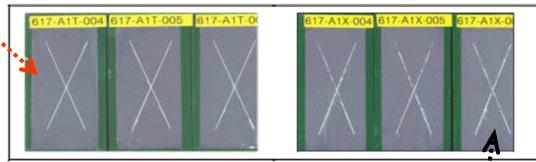


Figure 2: Complete Non-Chrome Systems T and X – 2000 Hours Salt Spray Exposure Alodine 5200 + SICOPOXY 577-630 + Deft 03-GY-321(T) / Deft 99-GY-001(X).

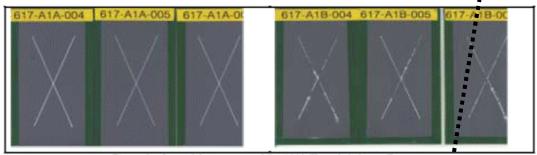


Figure 3: Control Systems A and B - 2000-Hour Salt Spray Exposure Alodine 1200S + Deft 02-Y-40 + Deft 03-GY-321(A)/Deft 99-GY-001(B).

 Similar stack up with Deft 99-GY-001 APC (System X) was next best system but not quite as good as Deft 33-GY-321 stack up



# AFRL Non-Chrome Coatings Evaluation: Brief History/Overview



# Phase IV Test Results - Filiform Corrosion

 The only coating stack up that exhibited performance comparable to the Cr-containing control was System T

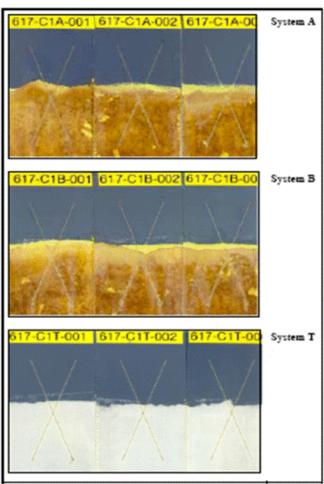


Figure 10: Filiform Corrosion - Control Systems A and B and Non-chrome System I.



# AFRL Non-Chrome Coatings Evaluation: Brief History/Overview



#### **Phase IV Results Summary**

Best Performing Coating = System T

Conversion Coat Primer Topcoat

Alodine 5200/5700 Sicopoxy 577-630 MIL-PRF-85285 (Deft 03-GY-321)

- Adhesion Data
  - Provided comparable performance to chromated control systems
- ASTM B 117 Neutral Salt Fog Data
  - Provided corrosion protection comparable to the control chromated system [Alodine 1200S CCC + MIL-PRF-23377 (Deft 02-Y-40) primer
    - + MIL-PRF-85285 (Deft 99-GY-001 APC) topcoat]
- Filiform Corrosion Data
  - Provided protection comparable to the chromated controls



# **Evaluation of Sicopoxy Primer for a Chromium-free Coating System**



## **Additional Laboratory & Outdoor Exposure Testing**

- Evaluate the Cr-free coating system identified in prior laboratory testing (AFRL Team) likely to be used in service
  - Non-chromate pretreatment
  - Chromium-free primer
  - Advanced performance topcoat
- Perform additional laboratory testing (CTIO/UDRI)
  - Validate earlier results
  - Expand property data base
- Perform outdoor exposure testing (CPCO/Battelle)
  - Best performing coating system
  - Various substrates
  - Chromated control coating system



## **Evaluation of Sicopoxy Primer for a Chromium-free Coating System**



## **Additional Laboratory & Outdoor Tests**

#### Laboratory testing

- ✓ Adhesion
  - Cross-hatch and modified X
  - 2024-T3, 7075-T6 substrates
  - D.I. water, hydraulic fluid, JP8+100 jet fuel, lubricating oil
- √ Flexibility and Elongation
  - 2" & 1" mandrel mend at -51°C
  - GE reverse impact
- √ Salt Fog Exposure
  - ASTM B117
  - 2,000 and 4,000 hours

#### Outdoor Exposure

- ✓ Daytona Beach, FL
  - Battelle marine exposure site
  - 45° rack, one year exposure
  - 2024-T3, 7075-T6 substrates
  - Scribed panels





# **Evaluation of Sicopoxy Primer for a Chromium-free Coating System**



## **Laboratory Testing Results**

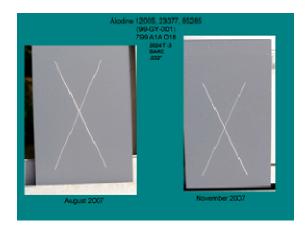
- Adhesion
- √ No failures with all Cr-free paint systems tested
- Low temperature flexibility
- ✓ Cr-free paint system selected passed this test
- GE reverse impact flexibility
- ✓ Cr-free paint system selected exhibited a relatively low % elongation but passed this test
- Salt fog exposure
- ✓ Minor/moderate corrosion product build up at 2,000 hr for Cr-free paint systems with Sicopoxy 577-630 primer: no change in rating at 4,000 hr
- ✓ No blistering observed
- ✓ Alodine 5200/5700 + Sicopoxy primer performed better than PreKote + Sicopoxy primer system
- ✓ Inhibiting system in the Sicopoxy primer remains effective over long periods of exposure

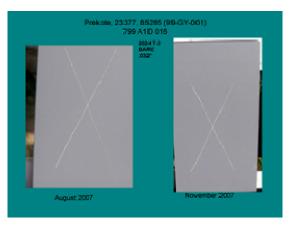


# **Evaluation of a Sicopoxy Primer** for a Chromium-free Coating System



## **Outdoor Exposure Testing Results**

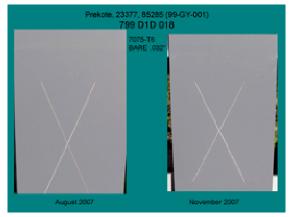






System 2024-T3: 85285 (99-GY-001) 3 & 6 Month Data









# **Evaluation of a Sicopoxy Primer for a Chromium-free Coating System**



## **Outdoor Exposure Testing Results**

#### • 3 & 6 months exposure

- ✓ Panels with Alodine 5700 and Sicopoxy primer performing better than those with the PreKote pretreatment and Sicopoxy primer
- ✓ Panels with Alodine 5700 and Sicopoxy primer showing comparable performance to the chromated control (Alodine 1200S/23377 primer) coating system
- √ Filiform corrosion not observed at this time

#### • 9 months exposure

- ✓ Panels with Alodine 5700 and Sicopoxy primer continue to perform better than those with PreKote pretreatment and Sicopoxy primer
- Outdoor exposure results correlate with the findings from the additional laboratory testing
- ✓ Test concluded after one year no change in relative rankings.

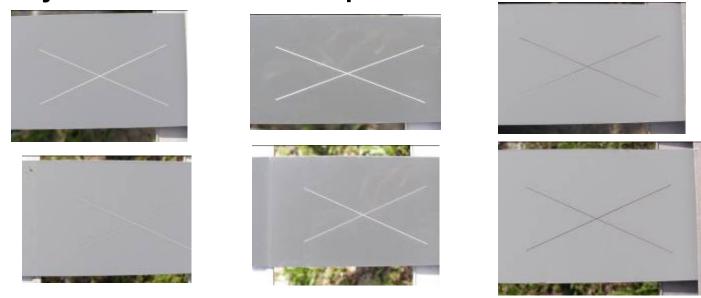
All results obtained indicate that this Cr-free paint system is ready to be tested in the field on small aircraft



# Alodine 5200/Sicopoxy/Deft ELT Non-Chrome Coating System



- Lab Testing indicates Alodine 5200/Sicopoxy/Deft ELT (APC)
   Nonchrome system performs the best of all Nonchrome Systems evaluated
- Daytona Beach outdoor exposure confirms that result



 AFRL/RXSSO, RXSSR, & RXSC working with AETC to conduct a field test of Alodine 5700/Sicopoxy/Deft ELT (APC) @ Randolph AFB



## **Sicopoxy Field Test Aircraft**



Aircraft Chosen: T-38 Talon

• Tail Number: 697085

• Flight Hours: ~15,000





## **Sicopoxy Test Aircraft**



Test section of the aircraft

Front view of the aircraft



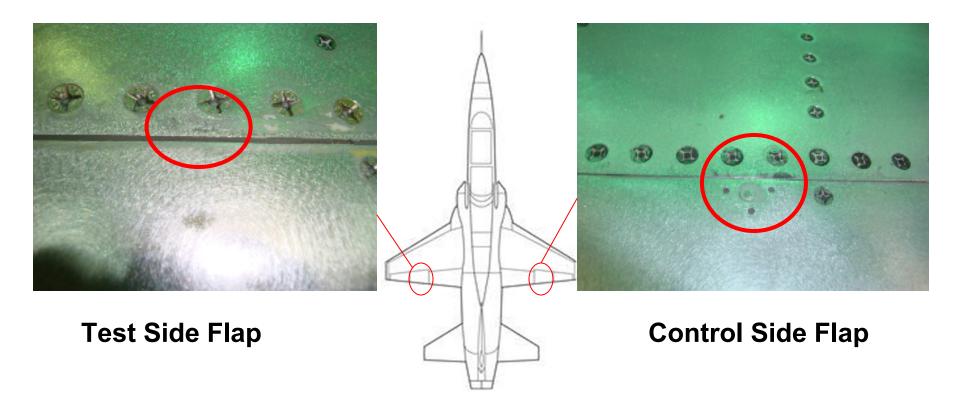


• First time we could evaluate condition of the aircraft before coating application.



#### **Corrosion On Aircraft Pre**





- Only two small areas of corrosion noted.
- On the topsides of right & left flaps ~ in the middle of the flaps.
- These are notoriously corrosion prone areas on aircraft.
- The larger area was on the chromated side of the aircraft.



## **Coating Systems**



#### Control

#### Non-Chrome

PreKote (3 Step Process)

**Brulin 815GD Cleaner** 

Alodine 5700 Nonchrome Conversion Coat

MIL-PRF-23377 Primer Sherwin Williams E90-G-203

ANAC 577-630 Non-Chrome Primer

Topcoats **Deft MIL-PRF-85285 (03-GY-308 and 03-GY-277)** 



### **Masked Areas**



- Magnesium
- Landing Gears
- Composite
- Sensors
- Engine
- Cockpit





## Aircraft Pretreatment Test Side



#### Wet the Aircraft Surface



#### Clean Step

- Spray and Scrub in Alkaline Cleaner with Scotch Brite pad
- Rinse off Alkaline Cleaner





#### Application of Alodine 5700

- Spray on Alodine 5700
- Rinse off Alodine
  - (2 to 5 Minute Dwell)







#### **Test Side Rinsate Collection**









- Alodine 5700 rinseate was collected, transferred to one 55 gal drum, and tested.
- Rinseate testing showed high Biological Oxygen Demand (BOD) and Chemical Oxygen Demand (COD).
- Total chrome in Alodine 5700 rinseate was 1.98 mg/L!



## Aircraft Pretreatment Control Side



#### •1st Application

Apply Prekote,Scrub in Prekote,Rinse off Prekote

#### •2<sup>nd</sup> Application

•Apply Prekote, Scrub in Prekote, Allow Prekote to Dry, Rinse

#### •3<sup>rd</sup> Application

Apply Prekote,Apply Prekote, Rinse off Prekote,





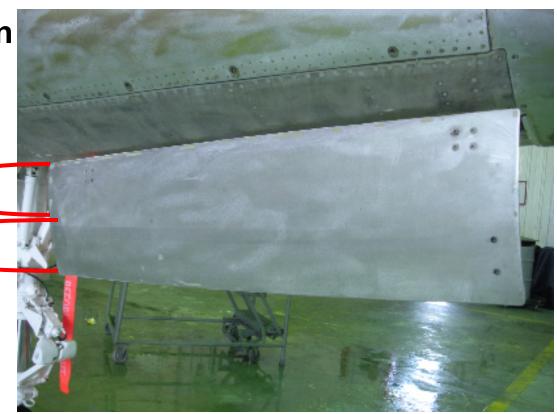
# Color Differences On Nose Landing Gear Door



Color differences between the two pretreatments were easily able to be seen on the nose landing gear door.

Standard Prekote Pretreatment

Alodine 5700 Pretreatment





#### **Pretreatment Observations**



- Alodine 5700 was applied correctly
- The surface was pre-cleaned using an alkaline wash and scotch-brite pads.
- The Alodine 5700 had a two to five minute dwell.
- No Powdering was observed on the aluminum, however application streaks were seen in the tail.
- Alodine 5700 held a water-break free surface better than the Prekote.
- Alodine 5700 gave a visible indication of application seen by the applicators.
- Primer was applied within 24 hours of Alodine 5700 application.



### **Primer Application**



#### **Control Side**



Standard chromated primer & process

#### **Test Side**





- Painters felt the Sicopoxy went on soother & easier than standard primer.
- Painters thought the Sicopoxy covered substrate much better than standard primer



### **Topcoat Application**



#### 1st Topcoat

- •Concerned that light grey color of Sicopoxy primer would make coating with light grey top coat difficult.
- However, since primer is semi-gloss & topcoat is gloss, it was easy to tell areas that hadn't been painted with topcoat.
- •Painters thought Sicopoxy was covered by topcoat much better than standard primer.



#### 2<sup>nd</sup> Topcoat

- Aircraft looked good.
- •No difference between control and test sides of the aircraft.





### **Final Inspection**









- Aircraft looked good.
- No visual difference between control and test side.
- Dry film thicknesses, color, & gloss were taken.
- Witness panels with both processes were taken for lab testing.



## **Sicopoxy Field Test Aircraft**



#### **Control Side**

#### **Test Side**



- Aircraft will be evaluated at 6 months and 1 year.
- Corrosion will be monitored by sensors at Randolph AFB & on the aircraft.





## **ELECTROACTIVE POLYMERS**



## Electroactive Polymers Background



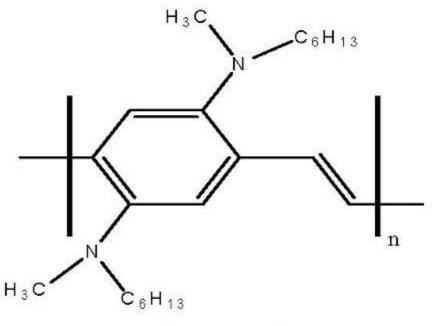
- Electroactive Polymers (EAPs) have been demonstrated under SERDP and ESTCP programs to inhibit corrosion on aluminum alloys as a pretreatment coating.
- EAPs performed similarly to chromate conversion coatings (CCC) when used with Cr(VI) primers, thus allowing for the removal of a significant amount of Cr(VI) from the coating system.
- Focus of this program is to demonstrate the viability of EAPs with both Cr(VI) and non-Cr(VI) primers to provide the military with a Cr(VI)-free coating system.
- These EAP coatings systems are attractive because they are a nonmetallic and offer a non hazardous alternative.
- The data from this program will be used to support the secondgeneration coatings using EAPs incorporated into military primers and removing the pretreatment requirement



#### **BAM-PPV**



 BAM-PPV Structure similar to polyaniline electroactive polymers (PAN)

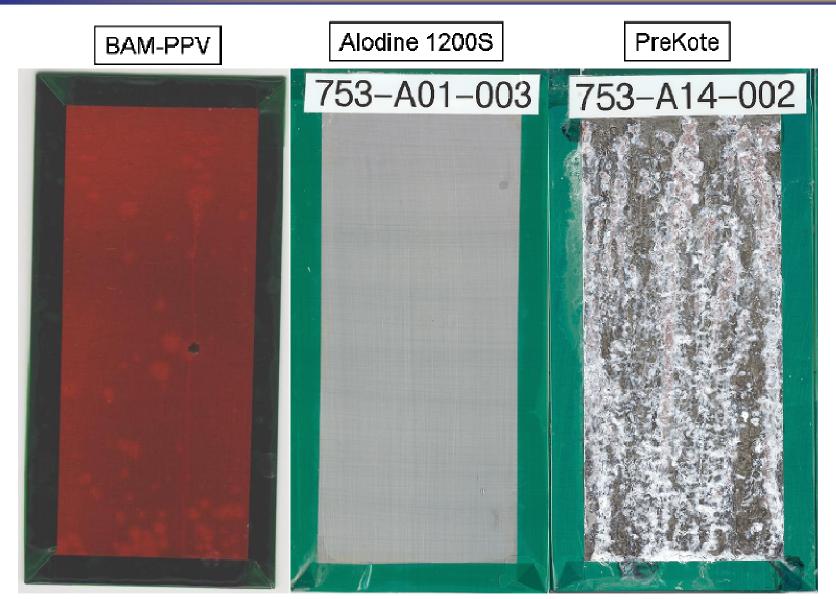


where n > 1

Poly(2,5-(bis-N-methyl-N-hexylamino)-p-phenylene vinylene), BAM-PPV











Alodine 1200S +
MIL-PRF-23377 chromated +
MIL-PRF-85285 APC

Alodine 1200S +
MIL-PRF-23377 non-chromated +
MIL-PRF-85285 APC









PreKote + MIL-PRF-23377 chromated + MIL-PRF-85285 APC PreKote + MIL-PRF-23377 non-chromated + MIL-PRF-85285 APC









BAM-PPV +
MIL-PRF-23377 chromated +
MIL-PRF-85285 APC

BAM-PPV +
MIL-PRF-23377 non-chromated +
MIL-PRF-85285 APC







## 20 months Outdoor Weathering



Alodine 1200S +
MIL-PRF-23377 chromated +
MIL-PRF-85285 APC

Alodine 1200S +
MIL-PRF-23377 non-chromated +
MIL-PRF-85285 APC







## 20 months Outdoor Weathering



PreKote +
MIL-PRF-23377 chromated +
MIL-PRF-85285 APC

PreKote +
MIL-PRF-23377 non-chromated +
MIL-PRF-85285 APC







## 20 months Outdoor Weathering



BAM-PPV +
MIL-PRF-23377 chromated +
MIL-PRF-85285 APC

753-A30-034

BAM-PPV +
MIL-PRF-23377 non-chromated +
MIL-PRF-85285 APC





# **EAP Field Test Flight Data on C-5 Door**



Test Area: Rear-Hatch Door





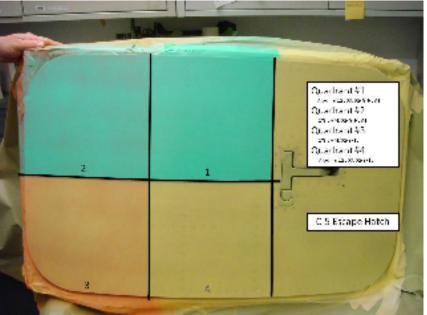
## **EAP Application to C-5 Door**



#### **Pretreatments**

#### **Primers**



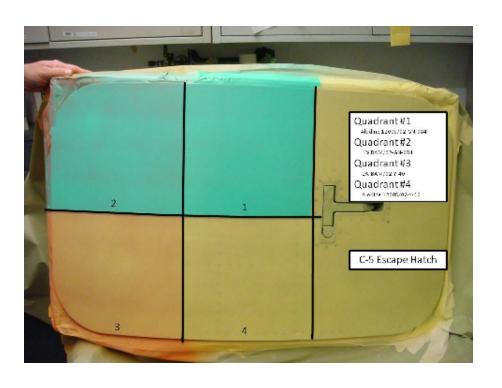




## **EAP Field Test Door Coating Test**Area Locations



2) BAM-PPV	1) Alodine 1600
Deft 02-Y-40	Deft 02-GN-084
Deft 99-GY-001	Deft 99-GY-001
3) BAM-PPV	4) Alodine 1600
Deft 02-Y-40	Deft 02-GN-084
Deft 99-GY-001	Deft 99-GY-001





## Field Test – Flight Data



- Goal is to minimize impact on 445 MXS/MGMFS schedules and operations
- Data will be available to stakeholders during testing and post-test
- Final report for AFRL involvement will be available to stakeholders
- Cumulative report to ESTCP with NAVAIR, ARL, and AFRL data will be available to stakeholders after final approval from ESTCP



## **Summary**



Mg Rich Primer

Sicopoxy

EAP Pretreatment



## **Questions?**





Bill Hoogsteden william.hoogsteden@wpaf.af.mil